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(4a*R*,8a*R*)-2,3-Diphenyl-4a,5,6,7,8,8a-hexahydroquinoxaline

Guo-Xi Wang and Heng-Yun Ye*

Ordered Matter Science Research Center, College of Chemistry and Chemical Engineering, Southeast University, Nanjing 210096, People's Republic of China
Correspondence e-mail: hyye@seu.edu.cn

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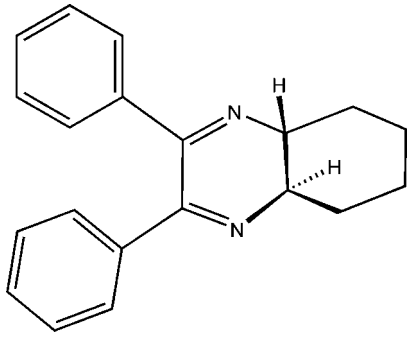
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.056; wR factor = 0.132; data-to-parameter ratio = 10.7.

The title molecule, $C_{20}H_{20}N_2$, is chiral; the absolute configuration follows from the known chirality of the input reagents. In addition to van der Waals forces, C—H$\cdots\pi$ ring interactions are also present. The angle between the planes of the phenyl rings is 65.6 (1)°. The heterocyclic ring of the quinoxaline system has a twist-boat configuration, while the cyclohexane ring has a chair configuration.

Related literature

For examples of the synthesis of non-centrosymmetric solid materials by reactions of chiral organic ligands and inorganic salts, see: Qu *et al.* (2004). For the geometric parameters of C=N bonds, see: Figuet *et al.* (2001); Kennedy & Reglinski (2001).



Experimental

Crystal data

$C_{20}H_{20}N_2$
M_r = 288.38
Orthorhombic, $P2_12_12_1$
a = 5.6253 (11) Å
b = 15.402 (3) Å
c = 18.315 (4) Å
V = 1586.8 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 293 (2) K
0.12 × 0.08 × 0.05 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.901, T_{max} = 1.000
(expected range = 0.898–0.996)
15676 measured reflections
2134 independent reflections
1880 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.132
S = 1.19
2134 reflections
200 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Table 1
D—H$\cdots\pi$-ring interactions calculated by *PLATON* (Spek, 2003).

*Cg*1 and *Cg*2 are the centroids of the phenyl rings C15–C20 and C8–C13, respectively.

D–H$\cdots Cg$	D—H	H$\cdots Cg$	$D\cdots Cg$	D—H$\cdots Cg$
C3—H3$A\cdots Cg1^{i}$	0.97	2.82	3.761 (4)	164
C4—H4$A\cdots Cg2^{ii}$	0.97	2.94	3.840 (3)	154
C11—H11$A\cdots Cg1^{iii}$	0.93	2.87	3.769 (3)	164

Symmetry codes: (i) $-\frac{1}{2}+x, \frac{3}{2}-y, -z$; (ii) $2-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iii) $\frac{1}{2}+x, \frac{1}{2}-y, -z$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FB2074).

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supplementary materials

Acta Cryst. (2008). E**64**, o359 [**doi:10.1107/S1600536807067505**]

(4a*R*,8a*R*)-2,3-Diphenyl-4a,5,6,7,8,8a-hexahydroquinoxaline

G.-X. Wang and H.-Y. Ye

Comment

Presence of chiral centres in organic ligands is important for synthesis of chiral coordination polymers (Qu *et al.*, 2004). We report here the crystal structure of (4a*R*,8a*R*)-4a,5,6,7,8,8a-hexahydro-2,3-diphenylquinoxaline (Fig. 1).

The lengths of the C═N double bonds (1.276 (3) and 1.278 (3) Å) are similar as in the following compounds containing the C═N double bonds: tris[(5-bromosalicylidene)aminoethyl]amine (Figuet *et al.* (2001) and *N*,*N*'-bis(salicylidene)-1,4,butanediamine (Kennedy *et al.*(2001).

Experimental

Benzil (2.10 g, 10.0 mmol) and (1*R*,2*R*)-(-)-diaminocyclohexane (1.20 g, 10.5 mmol) were dissolved in methanol (20 ml) containing sulfuric acid (0.08 g) as a catalytic agent. The solution was stirred at room temperature. After 4 h, a yellow precipitate appeared. It was filtered off and washed with chilled methanol (10 ml). The crude product was recrystallized by slow evaporation of the saturated ethanol solution. Yellow block-like crystals with dimensions of tenths of mm were isolated.

Refinement

All the H atoms could be found in the difference Fourier maps. Nevertheless, they were placed into the idealized positions and refined in a riding atom approximation with following constraints: $C_{methine}$—$H_{methine}$ = 0.98; $C_{methylene}$—$H_{methylene}$ = 0.97; C_{aryl}—H_{aryl} = 0.93 Å; U_{iso}H = 1.2U_{eq}C in all the cases. In the absence of significant anomalous scattering effects, 1531 Friedel pairs were merged. The absolute configuration was determined by synthesis. The chiral reactant (1*R*,2*R*)-(-)-diaminocyclohexane was used.

Figures

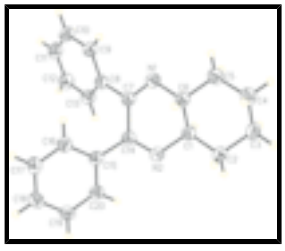

Fig. 1. A view of the title compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

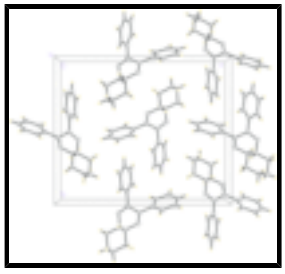

Fig. 2. The crystal packing of the title compound viewed along the *a* axis.

(4a*R*,8a*R*)-2,3-Diphenyl-4a,5,6,7,8,8a-hexahydroquinoxaline

Crystal data

$C_{20}H_{20}N_2$	F_{000} = 616
M_r = 288.38	D_x = 1.207 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 3447 reflections
a = 5.6253 (11) Å	θ = 3.4–27.5º
b = 15.402 (3) Å	μ = 0.07 mm^{-1}
c = 18.315 (4) Å	T = 293 (2) K
V = 1586.8 (5) Å^3	Block, yellow
Z = 4	0.12 × 0.08 × 0.05 mm

Data collection

Rigaku SCXmini diffractometer	2134 independent reflections
Radiation source: fine-focus sealed tube	1880 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.053
T = 293(2) K	θ_{max} = 27.6º
ω scans	θ_{min} = 3.5º
Absorption correction: multi-scan (CrystalClear; Rigaku, 2005)	h = −7→7
T_{min} = 0.901, T_{max} = 1.000	k = −19→20
15676 measured reflections	l = −23→23

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.056	H-atom parameters constrained
$wR(F^2)$ = 0.132	$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2 + 0.1683P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.19	$(\Delta/\sigma)_{max} < 0.001$
2134 reflections	$\Delta\rho_{max}$ = 0.13 e Å^{-3}
200 parameters	$\Delta\rho_{min}$ = −0.13 e Å^{-3}

80 constraints

Primary atom site location: structure-invariant direct methods

Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.010 (2)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7959 (6)	0.65882 (17)	0.09928 (14)	0.0512 (7)
H1A	0.6841	0.6288	0.1316	0.061*
C2	0.7195 (7)	0.75311 (18)	0.09215 (16)	0.0645 (8)
H2A	0.5568	0.7557	0.0748	0.077*
H2B	0.8198	0.7822	0.0567	0.077*
C3	0.7381 (7)	0.7995 (2)	0.16551 (17)	0.0699 (9)
H3A	0.7004	0.8605	0.1589	0.084*
H3B	0.6221	0.7752	0.1989	0.084*
C4	0.9823 (7)	0.79159 (18)	0.19858 (17)	0.0659 (9)
H4A	0.9824	0.8180	0.2467	0.079*
H4B	1.0958	0.8227	0.1685	0.079*
C5	1.0578 (7)	0.69734 (16)	0.20496 (15)	0.0592 (8)
H5A	1.2196	0.6944	0.2231	0.071*
H5B	0.9556	0.6678	0.2396	0.071*
C6	1.0429 (5)	0.65242 (16)	0.13156 (14)	0.0511 (7)
H6A	1.1540	0.6811	0.0982	0.061*
C7	1.0503 (5)	0.51148 (15)	0.08549 (13)	0.0476 (6)
C8	1.1020 (5)	0.41698 (17)	0.09128 (14)	0.0503 (7)
C9	1.3120 (6)	0.39001 (19)	0.12512 (15)	0.0583 (7)
H9A	1.4192	0.4307	0.1430	0.070*
C10	1.3595 (7)	0.3016 (2)	0.13178 (17)	0.0673 (9)
H10A	1.4989	0.2834	0.1544	0.081*
C11	1.2019 (7)	0.24103 (19)	0.10512 (18)	0.0684 (9)
H11A	1.2349	0.1821	0.1094	0.082*
C12	0.9971 (7)	0.26799 (18)	0.07241 (16)	0.0656 (9)
H12A	0.8894	0.2270	0.0552	0.079*
C13	0.9474 (6)	0.35524 (17)	0.06444 (15)	0.0565 (7)
H13A	0.8089	0.3725	0.0408	0.068*
C14	0.9184 (5)	0.54805 (16)	0.02031 (14)	0.0464 (6)

C15	0.9435 (5)	0.50996 (15)	−0.05353 (13)	0.0450 (6)
C16	1.1486 (5)	0.46658 (17)	−0.07485 (15)	0.0541 (7)
H16A	1.2679	0.4558	−0.0409	0.065*
C17	1.1760 (6)	0.43947 (19)	−0.14618 (15)	0.0601 (7)
H17A	1.3142	0.4108	−0.1601	0.072*
C18	1.0006 (6)	0.45455 (18)	−0.19673 (15)	0.0600 (8)
H18A	1.0213	0.4365	−0.2448	0.072*
C19	0.7950 (6)	0.49611 (19)	−0.17673 (15)	0.0578 (7)
H19A	0.6759	0.5059	−0.2110	0.069*
C20	0.7659 (6)	0.52341 (16)	−0.10515 (14)	0.0511 (6)
H20A	0.6258	0.5511	−0.0915	0.061*
N1	1.1133 (5)	0.56069 (14)	0.13814 (12)	0.0557 (6)
N2	0.7915 (5)	0.61639 (14)	0.02727 (12)	0.0526 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0596 (17)	0.0478 (14)	0.0463 (13)	0.0055 (13)	0.0037 (13)	−0.0004 (11)
C2	0.084 (2)	0.0535 (16)	0.0564 (16)	0.0211 (17)	−0.0011 (17)	−0.0020 (12)
C3	0.094 (3)	0.0526 (16)	0.0631 (18)	0.0175 (18)	0.0079 (19)	−0.0023 (13)
C4	0.091 (3)	0.0436 (14)	0.0631 (17)	−0.0021 (17)	0.0042 (18)	−0.0010 (13)
C5	0.077 (2)	0.0467 (14)	0.0543 (15)	−0.0056 (16)	−0.0053 (15)	0.0005 (12)
C6	0.0606 (17)	0.0410 (13)	0.0517 (14)	0.0015 (13)	0.0003 (14)	0.0025 (11)
C7	0.0542 (15)	0.0415 (12)	0.0471 (13)	0.0037 (12)	−0.0003 (12)	0.0030 (10)
C8	0.0596 (17)	0.0469 (13)	0.0445 (13)	0.0065 (13)	0.0057 (13)	0.0069 (11)
C9	0.0608 (18)	0.0550 (15)	0.0590 (16)	0.0095 (15)	−0.0028 (14)	0.0039 (13)
C10	0.078 (2)	0.0612 (18)	0.0628 (18)	0.0265 (18)	0.0031 (18)	0.0132 (14)
C11	0.095 (3)	0.0447 (14)	0.0657 (18)	0.0139 (18)	0.0167 (19)	0.0102 (14)
C12	0.085 (2)	0.0454 (14)	0.0665 (18)	−0.0041 (16)	0.0073 (18)	0.0080 (13)
C13	0.0631 (18)	0.0472 (13)	0.0592 (16)	0.0006 (14)	−0.0009 (15)	0.0066 (12)
C14	0.0490 (14)	0.0417 (12)	0.0486 (13)	−0.0007 (12)	−0.0001 (11)	0.0031 (10)
C15	0.0494 (14)	0.0380 (11)	0.0477 (13)	−0.0027 (12)	0.0028 (11)	0.0051 (10)
C16	0.0528 (16)	0.0557 (15)	0.0537 (14)	0.0044 (13)	0.0025 (13)	0.0015 (12)
C17	0.0623 (18)	0.0584 (16)	0.0596 (16)	0.0021 (15)	0.0103 (15)	−0.0048 (14)
C18	0.079 (2)	0.0521 (15)	0.0490 (14)	−0.0030 (16)	0.0042 (15)	−0.0046 (12)
C19	0.0713 (19)	0.0506 (14)	0.0513 (14)	−0.0014 (15)	−0.0106 (15)	0.0051 (12)
C20	0.0562 (16)	0.0431 (13)	0.0539 (14)	0.0002 (13)	−0.0018 (13)	0.0043 (11)
N1	0.0655 (15)	0.0453 (11)	0.0563 (13)	0.0060 (12)	−0.0078 (12)	0.0012 (10)
N2	0.0574 (14)	0.0507 (12)	0.0499 (12)	0.0069 (12)	−0.0034 (11)	0.0012 (10)

Geometric parameters (Å, °)

C1—N2	1.472 (3)	C9—C10	1.393 (4)
C1—C6	1.513 (4)	C9—H9A	0.9300
C1—C2	1.520 (4)	C10—C11	1.377 (5)
C1—H1A	0.9800	C10—H10A	0.9300
C2—C3	1.525 (4)	C11—C12	1.363 (5)
C2—H2A	0.9700	C11—H11A	0.9300
C2—H2B	0.9700	C12—C13	1.380 (4)

C3—C4	1.506 (6)	C12—H12A	0.9300
C3—H3A	0.9700	C13—H13A	0.9300
C3—H3B	0.9700	C14—N2	1.278 (3)
C4—C5	1.517 (4)	C14—C15	1.481 (3)
C4—H4A	0.9700	C15—C16	1.389 (4)
C4—H4B	0.9700	C15—C20	1.391 (4)
C5—C6	1.514 (4)	C16—C17	1.380 (4)
C5—H5A	0.9700	C16—H16A	0.9300
C5—H5B	0.9700	C17—C18	1.373 (5)
C6—N1	1.472 (3)	C17—H17A	0.9300
C6—H6A	0.9800	C18—C19	1.372 (5)
C7—N1	1.276 (3)	C18—H18A	0.9300
C7—C8	1.488 (3)	C19—C20	1.387 (4)
C7—C14	1.514 (3)	C19—H19A	0.9300
C8—C13	1.379 (4)	C20—H20A	0.9300
C8—C9	1.397 (4)		
N2—C1—C6	109.6 (2)	C13—C8—C7	121.7 (3)
N2—C1—C2	110.0 (2)	C9—C8—C7	119.2 (3)
C6—C1—C2	110.8 (3)	C10—C9—C8	119.5 (3)
N2—C1—H1A	108.8	C10—C9—H9A	120.3
C6—C1—H1A	108.8	C8—C9—H9A	120.3
C2—C1—H1A	108.8	C11—C10—C9	120.5 (3)
C1—C2—C3	110.7 (2)	C11—C10—H10A	119.7
C1—C2—H2A	109.5	C9—C10—H10A	119.7
C3—C2—H2A	109.5	C12—C11—C10	119.6 (3)
C1—C2—H2B	109.5	C12—C11—H11A	120.2
C3—C2—H2B	109.5	C10—C11—H11A	120.2
H2A—C2—H2B	108.1	C11—C12—C13	120.9 (3)
C4—C3—C2	112.2 (3)	C11—C12—H12A	119.5
C4—C3—H3A	109.2	C13—C12—H12A	119.5
C2—C3—H3A	109.2	C8—C13—C12	120.4 (3)
C4—C3—H3B	109.2	C8—C13—H13A	119.8
C2—C3—H3B	109.2	C12—C13—H13A	119.8
H3A—C3—H3B	107.9	N2—C14—C15	118.1 (2)
C3—C4—C5	111.3 (3)	N2—C14—C7	120.1 (2)
C3—C4—H4A	109.4	C15—C14—C7	121.7 (2)
C5—C4—H4A	109.4	C16—C15—C20	118.5 (2)
C3—C4—H4B	109.4	C16—C15—C14	121.8 (2)
C5—C4—H4B	109.4	C20—C15—C14	119.6 (2)
H4A—C4—H4B	108.0	C17—C16—C15	120.3 (3)
C6—C5—C4	110.7 (2)	C17—C16—H16A	119.9
C6—C5—H5A	109.5	C15—C16—H16A	119.9
C4—C5—H5A	109.5	C18—C17—C16	120.5 (3)
C6—C5—H5B	109.5	C18—C17—H17A	119.8
C4—C5—H5B	109.5	C16—C17—H17A	119.8
H5A—C5—H5B	108.1	C19—C18—C17	120.3 (3)
N1—C6—C1	110.0 (2)	C19—C18—H18A	119.8
N1—C6—C5	110.5 (2)	C17—C18—H18A	119.8
C1—C6—C5	111.6 (2)	C18—C19—C20	119.6 (3)

N1—C6—H6A	108.2	C18—C19—H19A	120.2
C1—C6—H6A	108.2	C20—C19—H19A	120.2
C5—C6—H6A	108.2	C19—C20—C15	120.9 (3)
N1—C7—C8	118.2 (2)	C19—C20—H20A	119.6
N1—C7—C14	120.7 (2)	C15—C20—H20A	119.6
C8—C7—C14	121.1 (2)	C7—N1—C6	115.7 (2)
C13—C8—C9	119.1 (3)	C14—N2—C1	116.5 (2)

D—H···π-ring interactions calculated by PLATON (Spek, 2003)

D–H···*Cg*	D—H	H···*Cg*	D···*Cg*	D—H···*Cg*
C3—H3A···*Cg*1^{i}	0.97	2.82	3.761 (4)	164
C4—H4A···*Cg*2^{ii}	0.97	2.94	3.840 (3)	154
C11—H11A···*Cg*1^{iii}	0.93	2.87	3.769 (3)	164

Symmetry codes:(i) −1/2 + *x*,3/2 − *y*,-*z*; (ii) 2 − *x*,1/2 + *y*,1/2 − *z*; (iii) 1/2 + *x*,1/2 − *y*,-*z*. *Cg*1 and *Cg*2 are the centroids of the phenyl rings C15–C20 C8–C13, respectively.

Fig. 1

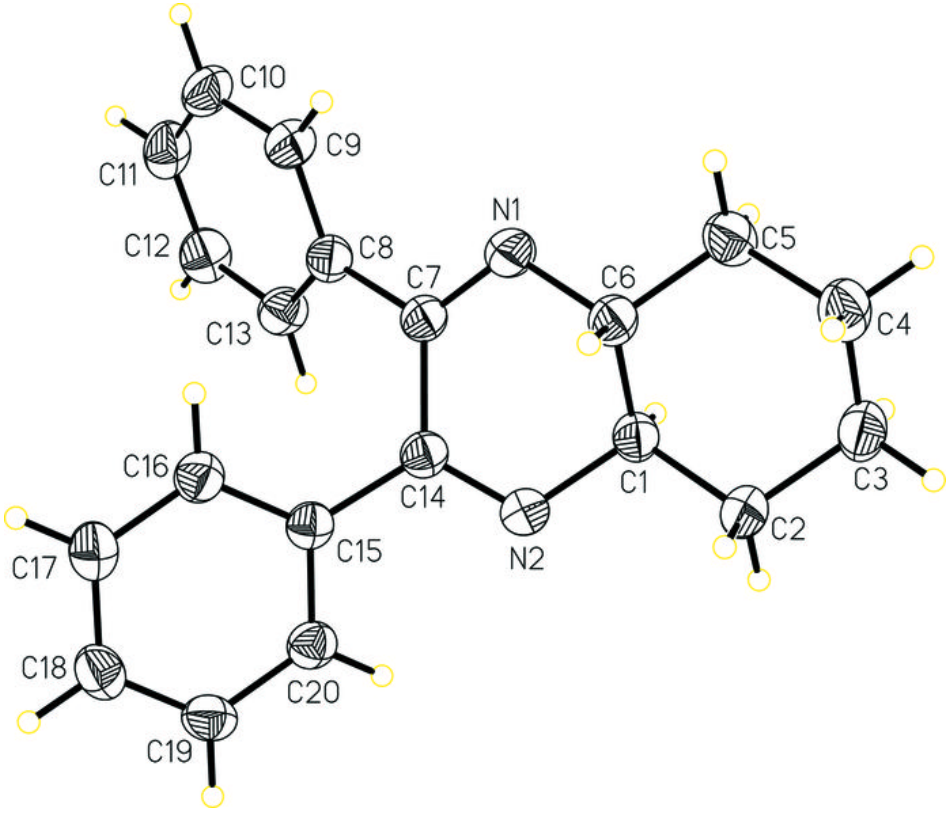
C10
C9
C11
N1
C12
C8
C7
C6
C5
C13
C4
C16
C14
C1
C3
C15
N2
C2
C17
C18
C20
C19

Fig. 2